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Nitrogen Trifluoride Combustion and Suppression

J. R. WYATT AND J. J. DECORPO

*Chemical Diagnostics Branch
Chemistry Division*

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NITROGEN TRIFLUORIDE COMBUSTION AND SUPPRESSION

INTRODUCTION

This report discusses the work carried out by NRL Code 6112 for the period 1 October 1979 to 30 September 1980 on NAVSEA PMS 405's project concerning NF_3 combustion and suppression. A previous report discussed the initial work on this project [1]. Early in the program the hazardous nature of the experiments became evident; thus, a decision was made to conduct the larger scale experiments at a facility specifically designed for such work. Southwest Research Institute was chosen because of its ample facilities and its excellent work for NRL (Code 6112) on a previous project involving explosive/flammability studies for other NAVSEA (Codes 921 and 05R14) projects.

There were three primary objectives of this program. The first objective was to determine the effectiveness of halon 1301 in preventing combustion of ethylene in an NF_3 -enriched air atmosphere. The second objective was to measure the severity of the reaction that develops when NF_3 -enriched air/ethylene mixtures are initiated with or without the presence of halon 1301. The third objective was to determine if any toxic compounds are produced during an NF_3 /ethylene fire suppressed with halon 1301. The approach was based on successful techniques developed on previous programs [2-4]. In the following paragraphs, the approach to accomplishing the above-stated objectives is described.

BACKGROUND

In the preparation for this study, the literature was surveyed to find the flammability characteristics of ethylene and the characteristics of NF_3 and halon 1301 [5-11]. The flammability characteristics of ethylene in air and other oxidizers are summarized in Table 1. The flammability limits of ethylene in air are 2.7% to 36% by volume. In a nitrous oxide-enriched air atmosphere, the flammability limits are somewhat wider, 1.9% to 40%. Presumably the effect of NF_3 on the flammability limits of ethylene would be the same; that is, it would widen the envelope of flammable concentrations. Craven and Foster [10] performed experiments on ethylene/air mixtures with excess nitrogen in a spherical vessel at elevated pressures. Their results are shown in Fig. 1. Note in this figure that the effect of increasing the initial pressure is to increase the upper flammable limit, while the lower limit appears to be unchanged. Additionally, the effect of excess nitrogen is to decrease the upper flammable limit, again with the lower flammable limit remaining virtually unchanged.

Table 1 — Flammability Characteristics of Ethylene

Oxidizer	Flammability Limits (%)	P_{\max} (atm)	Chamber	Reference
Air	2.7 — 36.0	—	Cylindrical	2
Air	3.02 — 34.0	—	Cylindrical	3
Air	--	12.0	—	4
Oxygen	2.9 — 80.0	—	Cylindrical	2
Nitrous Oxide	1.9 — 40.0	—	Cylindrical	2

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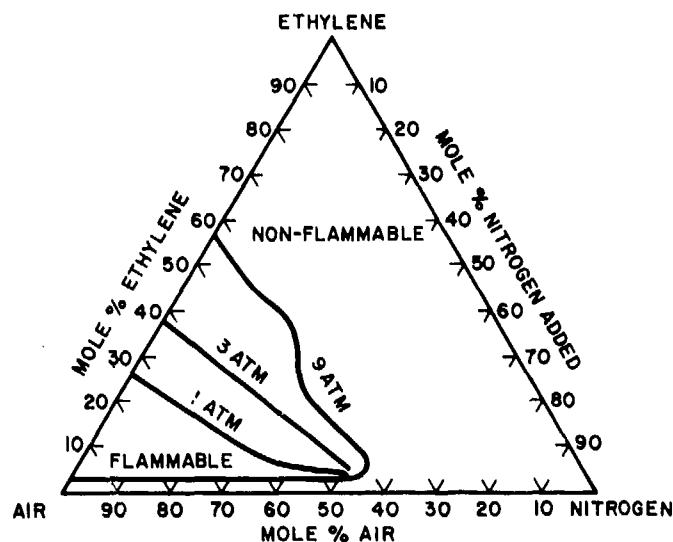


Fig. 1 — Flammability of ethylene in air with added nitrogen

The following is an example of using Fig. 1 to determine the flammability of an ethylene-air-nitrogen mixture. Assume for this example that the concentration ratio of air:ethylene:nitrogen is 7:3:3. Thus the mole % ethylene to air is $100\% \times 3/(7+3) = 30\%$; likewise the mole % air to nitrogen is $100\% \times 7/(7+3) = 70\%$. Therefore, draw a line from 30 on the left-hand side of the triangle to the opposite vertex. Do the same from 70 on the bottom side to the top vertex. The intersection of these lines is in a region just flammable at 3 atm (300 kPa) total pressure but outside the flammability region at 1 atm (100 kPa) pressure. Alternatively, this diagram can be used to determine what amount of nitrogen is necessary to inert an air-ethylene mixture having a ratio of 8:2. Draw a line from 20% on the left-hand edge to the opposite vertex. Draw a line from the top vertex through the point where the other line intersected the flammability region to the bottom. This shows that when the mole % of air is less than 80% compared to nitrogen the mixture is not flammable.

Halon 1301 has been used for years as an extinguishing agent for fires from flammable vapors and liquids. A review of the literature showed that ethylene/air flames are extinguished by halon 1301 concentrations of 5.1% to 6.5% by volume [10,11]. These extinguishment values were obtained in an Imperial Chemical Industries (ICI) cup burner. In this device, the air around a steady flame is flooded with halon 1301 until the flame is extinguished.

The Bureau of Mines has performed some tests to determine the effectiveness of halon 1301 in the suppression of methane/air explosions [10]. In these tests, the researchers found that the methane/air explosions were effectively quenched by halon 1301 only when the agent was applied before the fireball exceeded about 46 cm (18 in.). Dispersion experiments revealed that extinguishment failures in the mine tests were attributed to nonuniform agent distribution, which resulted in the formation of pockets of low halon concentration. Additionally, the researchers found that the rate of halon delivery and the turbulence caused by the halon delivery affected the quenching of the methane explosions. Thus, in the design of an explosion-suppression system one must consider not only the concentration of halon required to extinguish the flames but also the delivery rate and the delay time prior to the application of the agent.

One controversial characteristic of halon 1301 is its potential for thermal decomposition when applied to a fire. Some theories of extinguishment require this decomposition to produce bromine radicals which then tie up active intermediate combustion products into less active species. Whether required or not, decomposition does occur and has been observed to some degree in virtually every fire extinguished by halon 1301. The by-products are predominately hydrogen fluoride (HF) and hydrogen bromide (HBr) [10]. The toxicity of these by-products in combination with the NF_3 oxidizing agent is a potential problem, depending on the concentrations of the by-products which are produced.

FLAMMABILITY TESTS

Flammability Apparatus

The objective of the flammability tests was to study the ability of halon 1301 to extinguish ethylene flames in an NF_3 -enriched air atmosphere. The apparatus used to perform these experiments is called the ICI cup burner. This apparatus consists of a glass cup that is loosely packed with glass wool and located in the center of a vertical glass tube. Air containing metered quantities of NF_3 and ethylene is passed up through the tube and a flame is ignited on top of the burner. Once the flame has stabilized, a metered flow of halon 1301 is added to the main oxidizer and fuel stream. The halon 1301 is slowly added until the flame is extinguished.

A schematic drawing of the burner is shown in Fig. 2. The burner is a 2.8-cm (1.1-in.) diameter cup mounted at the top of a glass tube. A standard hemispherical joint is attached to the bottom of the tube, giving an overall length of 25.4 cm (10 in.). The burner is mounted at the axis of a glass chimney 8.4 cm (3.3 in.) in diameter, above a bed of glass beads. A mixture of air and agent passes through this bed and flows up the chimney and past the burner. The top of the burner is at a sufficient distance from the bed to permit the dispersion of any initial disturbances in the flow. It was found experimentally that 25.4 cm (10 in.) was adequate to accomplish this without unduly affecting the mechanical stability. The transition from the diameter of the tube to that of the burner is profiled so as to cause minimum disturbance to the flow. Nearly all flames on this burner are both smooth and motionless.

The flows of both air and halon vapor are measured by calibrated rotameters. Both streams combine in a glass T-piece, and mixing is completed in the bed of glass beads. The burner itself is connected through its hollow stem and a length of flexible tubing to a fuel source.

The operating procedure generally used for the typical case of a fuel in air is as follows:

1. Set an air rotameter at 40 l/min (1.4 ft³/min).
2. Set the fuel-flow rotameter at 0.5 l/min (0.018 ft³/min).
3. Light the fuel in the burner and put on the chimney.
4. Add the halon to the air supply in coarse rotameter increments of 1.0 l/min (0.035 ft³/min). Continue the addition of halon until extinction is achieved.
5. Turn off the halon flow and remove the chimney.
6. Light the fuel again and replace the chimney.

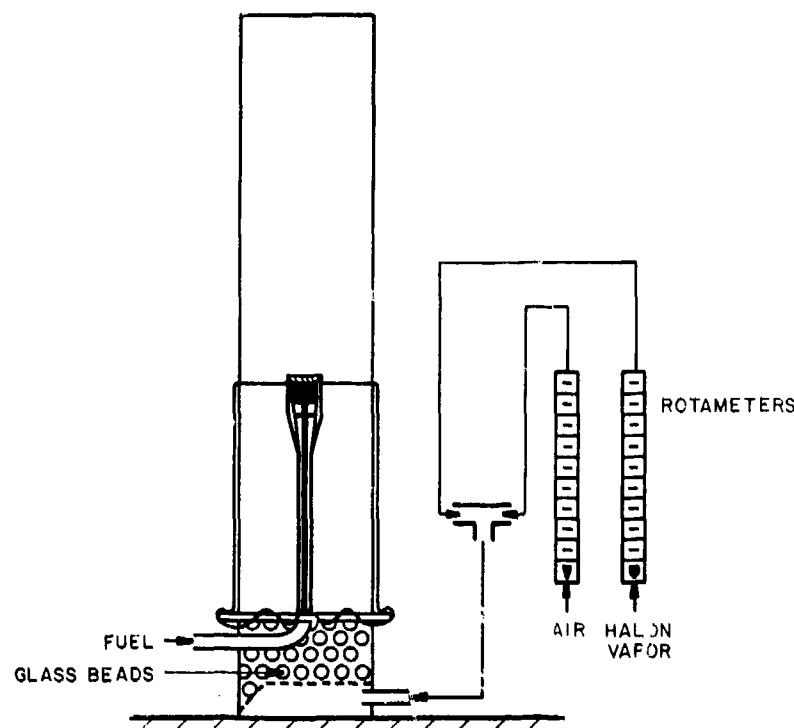


Fig. 2 -- Schematic diagram of ICI cup burner

7. Add the halon at one rotameter setting below the value observed at the extinction point measured in step 4.
8. Increase the halon by the fine rotameter increments of 1.0 l/min (0.035 ft³/min) until extinction is achieved.
9. Repeat the extinguishment tests until five identical extinction values are achieved.
10. Increase the fuel flow by 0.5 l/min (0.018 ft³/min) increments up to 5 l/min (0.18 ft³/min) and obtain extinguishment values at each stage.
11. Plot a graph of % v/v halon for extinguishment against fuel flow rate.
12. Obtain the extinction value from the peak value on the graph.

Flammability Test Results

The results of the flammability tests are summarized in Fig. 3. These tests consisted of flowing air-NF₃-ethylene-halon 1301 mixtures through the ICI cup burner. The air flow rate was constant at 40 l/min (1.4 ft³/min). The ethylene flow rate was held constant in any one experiment but was varied over a range of 0.06 to 0.6 l/min (0.002 to 0.02 ft³/min) in the course of the tests. Similarly, the NF₃ flow rate was constant in any one experiment, but it was eventually varied to give concentrations from 0 to 10% NF₃ in air. As shown in Fig. 3, the percent halon required for

extinction was relatively constant at each NF_3 concentration over the range of ethylene flow rates. For an unknown reason the 10% NF_3 mixture was particularly sensitive to ethylene flow rates lower than about 0.20 l/min (0.007 ft³/min). The information contained in Fig. 3 can be cross plotted to obtain the halon extinguishment concentration as a function of the NF_3 concentration. This is shown in Fig. 4. This figure shows the relative insensitivity of the extinguishment concentration of halon 1301 to variations in the ethylene flow rate. It indicates that about 6.5% halon is required to extinguish ethylene-air flames with no NF_3 enrichment. For every addition of 1% NF_3 , an average of 0.88% additional halon is required for extinguishment.

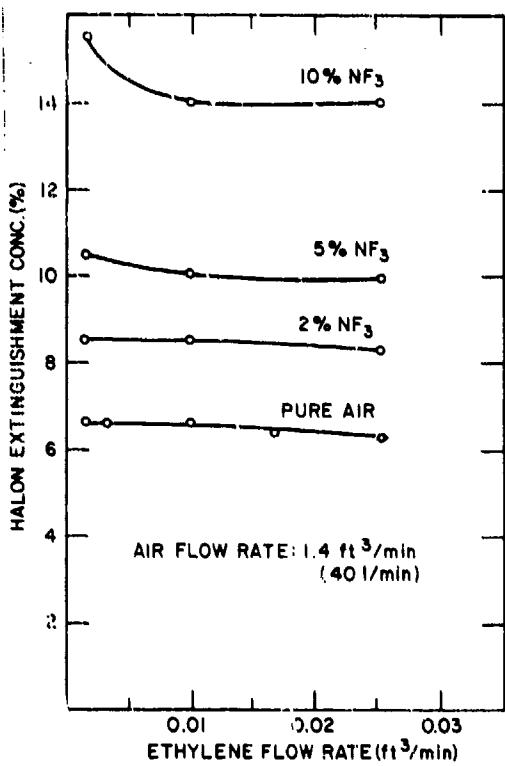


Fig. 3 -- Flammability study test results
(1 ft³/min = 28.3 l/min)

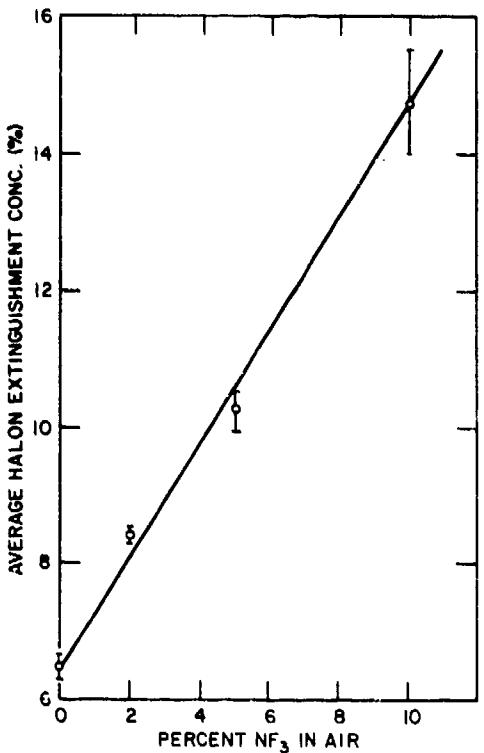


Fig. 4 -- Halon concentration required to extinguish ethylene flames as a function of NF_3 enrichment

EXPLOSIBILITY TESTS

Experimental Arrangements

The tests described in this section of the report were conducted at Southwest Research Institute's Energetic Materials Research Laboratory of the Department of Ballistics and Explosives Sciences. The test apparatus was housed in a protective cell, while the bulk of the instrumentation was located just outside of the cell. The experimental arrangements allowed the test gases to be mixed in one chamber and sampled for composition before being loaded into a detonation chamber and ignited. Pressure sensors and suitable signal conditioning recorded the severity of the explosion. Details of the experimental system including the detonation chamber, charging system, initiation, instrumentation, and data reduction are described in the following subsections.

Detonation Chamber

The detonation chamber used on this program consisted of a steel cylinder machined from bar stock. The inside diameter of the vessel was 5.08 cm (2.0 in.) with a wall thickness of 2.54 cm (1.0 in.). The internal length of the vessel had provisions for the installation of seven pressure transducers, spaced at equal intervals of 7.62 cm (3.0 in.) and arranged so that the pressure-sensing element was flush with the inside diameter of the vessel.

Ion probes were also installed in the vessel at 19.05 cm (7.5 in.) and 49.53 cm (19.5 in.) from the ignitor end. A portion of these probes extended into the interior of the chamber. Table 2 gives a list of instrument locations.

Table 2 — Position of Transducers
Relative to Ignitor End of the
Explosion Vessel

Transducer	Location	
	(cm)	(in.)
Pressure, P1	11.43	4.5
Pressure, P2	19.05	7.5
Pressure, P3	26.67	10.5
Pressure, P4	34.29	13.5
Pressure, P5	41.91	16.5
Pressure, P6	49.53	19.5
Pressure, P7	57.15	22.5
Pressure, P8	68.58	27.0
Ionization probe, I1	19.05	7.5
Ionization probe, I2	49.53	19.5
Temperature probe, T1	49.53	19.5

The end caps used on the vessels were machined from stainless-steel bar stock and attached to the cylindrical portion of the vessel with nine steel bolts. Pressure tightness is assured by use of a Bal-Seal Engineering high-pressure and high-temperature seal. The end caps were machined to accept a pressure transducer and a gas charging line or an initiator and a gas purging line.

Filling and Firing Sequence

The desired mixture to be initiated on each test was obtained by first evacuating the mixing and detonation and sampling chambers and then backfilling the mixing chamber with the various gases according to the method of partial pressures. The lowest percentage gas was introduced first and the highest percentage gas (air) was introduced last. The mixing chamber was filled to 5 or 10 atm (500 or 1000 kPa) and the mixture was agitated by a rotor inside the mixing chamber for a period of 5 min. After the completion of the mixing period, a sample of the mixed gases was introduced into a stainless-steel sampling vessel. The sampling vessel was filled to approximately 2 atm (200 kPa) to facilitate the gas analysis. After the sample was collected, the detonation chamber was filled to 1 atm (100 kPa), and the mixture was ignited with an exploding bridgewire (EBW). A schematic diagram of the experimental apparatus is given in Fig. 5.

Gas Analysis

The gas samples collected in the manner described in the previous section were transported immediately after ignition to the U.S. Army Fuels and Lubricants Laboratory, where the gas

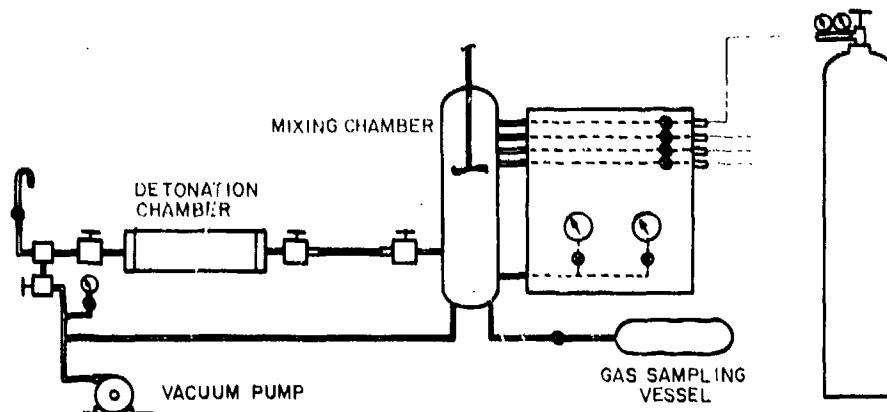


Fig. 5 — Schematic diagram of the gaseous explosion apparatus

analysis was performed. The infrared-absorption procedure for analyzing the gas samples was developed specifically for these experiments. The gas sample at roughly 2-atm (~ 200 -kPa) pressure was transferred to a 10.2-cm (4-in.)-path gas cell with potassium chloride windows, and an absorption spectrum was obtained in the 4000 cm^{-1} to 600 cm^{-1} region of the infrared with a Beckman Model 620 MX microprocessor controlled infrared spectrophotometer. The method requires calibration with known concentrations of the analyzed gases.

Blends of halon 1301, C_2H_4 , or NF_3 in air, in the range of approximately 2% to 20%, were run. Baseline technique was used to calculate the absorbance for concentration. Analytical frequencies were chosen for each gas that would not interfere with the other gases: 2290 cm^{-1} for halon 1301, 3090 cm^{-1} and 3140 cm^{-1} for C_2H_4 , and 1940 cm^{-1} for NF_3 . Background frequencies were also chosen: 2140 cm^{-1} and 2480 cm^{-1} for halon, 2890 cm^{-1} and 3290 cm^{-1} for C_2H_4 , and 1990 cm^{-1} and 1880 cm^{-1} for NF_3 . Calibration curves of absorbance vs concentration were plotted, and each gas was found to absorb linearly with respect to concentration.

Initiation

The gaseous mixtures were ignited using commercial exploding bridgewires (EBW). An EBW initiator consists of a fine gold wire 3.175 mm (0.125 in.) long mounted on an epoxy base. The gold wire is vaporized by applying a short-duration ($1\text{-}\mu\text{s}$) pulse. The energy is applied so fast that the wire material phase changes (melting and vaporization) are restricted by inertia. When the inertia is overcome, vaporization of the wire occurs as an explosion emitting thermal energy and a shock wave. The output of the initiator is essentially independent of the gas composition and initial pressure, which makes it an excellent initiator for this application.

A Reynolds Industries EBW firing module was used to pulse the EBW. At the same instant, the firing module generated an event trigger for the instrumentation system.

Instrumentation System

The purpose of the instrumentation system was to determine if a reaction occurred, to determine when it occurred, and to assess the relative severity of the reaction. For this purpose, seven pressure transducers were mounted in the cylindrical wall of the vessel and one in the center of

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the pressurizing head. The transducers were mounted so that their sensitive diaphragms were flush with the inside surface of the vessel. The pressure transducers used on this program were PCB instruments, models 102A02 and 102A03 piezoelectric gauges. These transducers have a sensitive surface or diaphragm that is displaced under an applied pressure. This pressure is transmitted by the diaphragm to an interior piezoelectric element, which responds with an output proportional to the applied pressure. The 102A02 transducers are designed for pressures up to 1000 psi (~7 MPa). The 102A03 transducers are designed for pressures up to 10,000 psi (~70 MPa). Both transducers are designed to minimize acceleration and transient temperature sensitivity.

Each pressure transducer was dynamically calibrated during this program using a drop-weight calibrator. This device employs a weight which is dropped onto a piston mounted in an oil-filled cavity. A PCB transducer and a Kulite reference transducer are mounted in the cavity. By comparing the output of the Kulite reference gauge and the test gauge for a wide variety of drop heights and weights, the sensitivity (in V/psi) of each transducer was determined.

A schematic diagram of the instrumentation system is given in Fig. 6. The output of the pressure transducers was recorded, after appropriate amplification, on a Honeywell 101 wideband tape recorder. In addition to the pressure-transducer output, signals from two ionization probes were recorded in the same way. Temperature measurements inside the vessel were made using a type K thermocouple and a strip-chart recorder.

Data Reduction

The data reduction for this program was performed with the aid of the Department of Explosives and Ballistics Sciences CAMAC (computer automated measurement and control) system. A data trace is reduced using this system by playing up to four channels at a time from the tape recorder into a Biomation 1015 four-channel transient recorder. The transient recorder digitizes

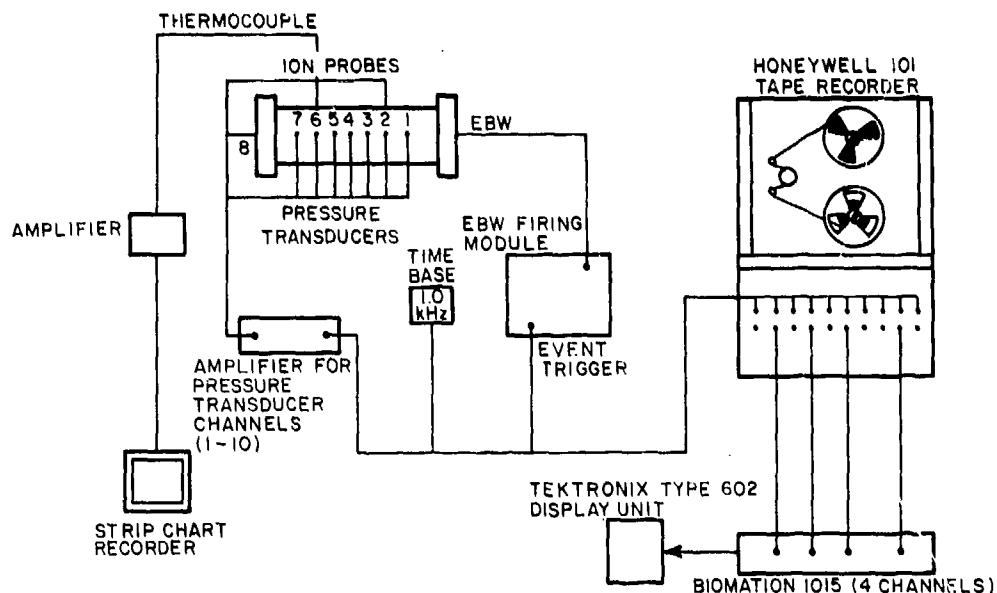


Fig. 6 — Schematic diagram of data acquisition system

and stores the incoming signals. Once the data are properly formatted in the transient recorders, the operator issues a command from the console which instructs the CAMAC crate to extract the digitized data from the memory of the transient recorder and transmits the data to a disk file on the PDP 11/70 minicomputer. In practice, calibration pulses are also digitized and transmitted on each test to facilitate conversion to engineering units. Using this system with the Biomation recorder, vertical resolution is 1/1024th of the full-scale voltage setting. Samples may be taken at intervals of 0.1 ms or greater, and all four channels are sampled simultaneously. A record consists of 1024 samples, so the duration of sampling is 1024 times the sample rate. Figure 7 shows a sample of the CAMAC data.

The initial rate of pressure rise and the impulse up to the peak pressure are estimated by another program. The pressure rate is determined by locating those samples on the initial pressure rise which are between 10% and 90% of the peak pressure. These points are then fitted into a second-order polynomial. The polynomial is differentiated, and the maximum slope is taken to be the pressure rise rate. The impulse is calculated simply by integrating the pressure records up to the time at which the maximum pressure is achieved.

Explosibility Test Results

The results of the explosibility tests are described in this section of the report. The peak pressure is obtained using the algorithm described in the section on data reduction. For ease of presentation, the time histories are grouped into three categories: ethylene-air tests, ethylene-NF₃-air tests, and ethylene-NF₃-halon 1301-air tests. Within each category, the tests are presented in order of increasing ethylene concentration.

Three types of reactions were observed in these experiments: no reaction, combustion, and deflagration. The criteria used to classify the reactions were developed on a previous Navy program [4] and are based solely on parameters inferred from the pressure history measured in the detonation vessel. The inferred parameters include the peak side-on pressure, the peak reflected pressure and the rate of pressure rise.

A test was classified as *no reaction* if no measurable increase in pressure was detected by the instrumentation system (either pressure or temperature).

Combustion reactions are typified by a gradually increasing, relatively smooth pressure trace. Also, in a combustion reaction no shock is transmitted, so all transducers should measure the same pressure at all times regardless of orientation within the chamber. Thus, the ratio of the reflected pressure to the side-on pressure, P_r/P_{so} , should be close to unity.* Also, the pressure pulse rate should be low, generally less than 3000 psi/s (20 MPa/s).

Deflagration reactions are typified by a sharp, nearly discontinuous increase in pressure. Unlike in combustion reactions, a definite shock wave is created, so the reflected pressure may greatly exceed the side-on pressure. The pressure rise rate in deflagration reactions generally is more than 3000 psi/s (20 MPa/s).

In this program the above criteria were sometimes inadequate to characterize a reaction as combustion or deflagration. The pressure traces were often more complicated than expected, often consisting of an initial pressure rise, a pressure decay, and an even faster pressure rise. It

*The reflected pressure is measured at Station 8 in the end cap of the vessel opposite the ignition source. The side-on pressure is measured at stations 1 to 7 at various points on the sidewall of the cylinder.

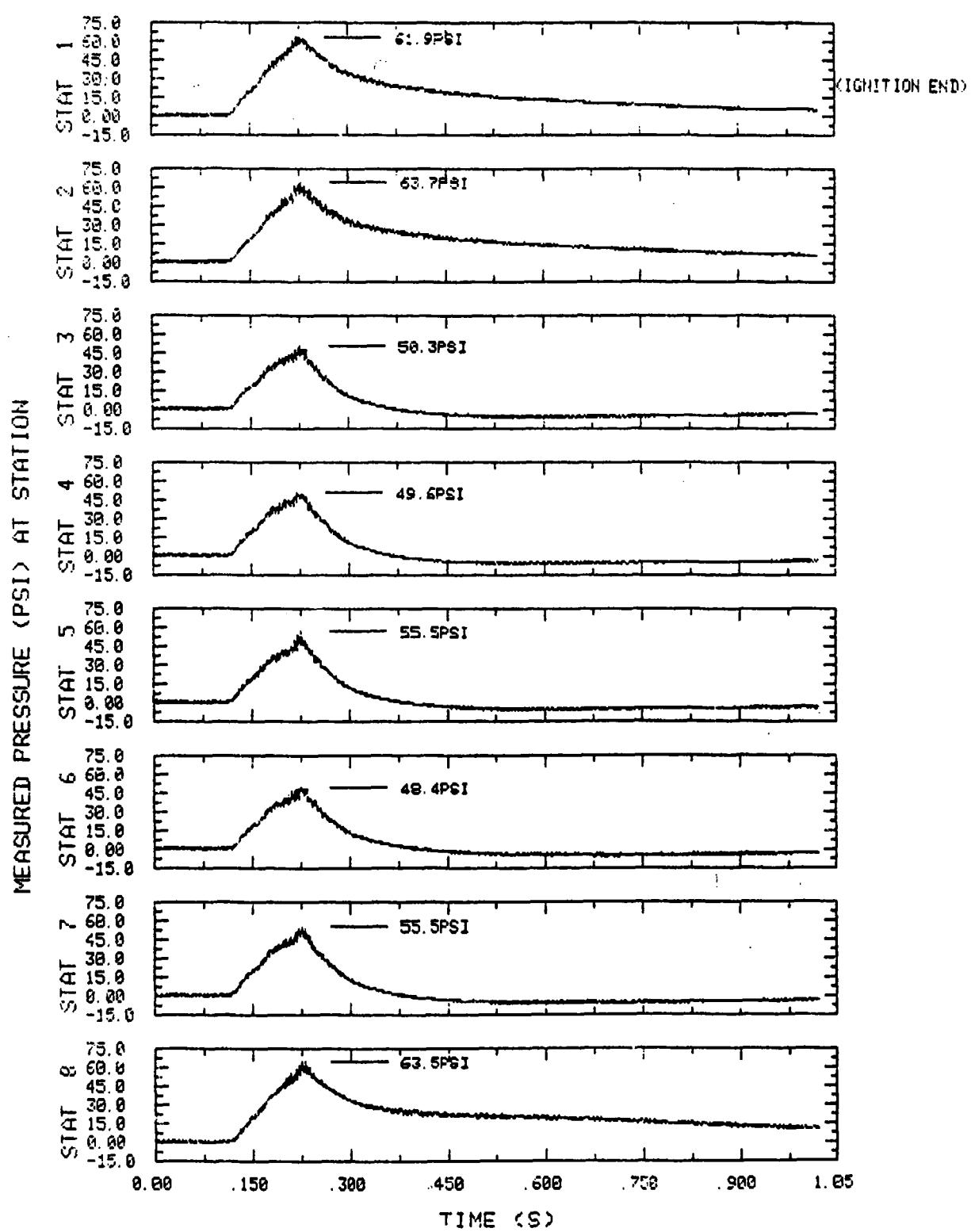


Fig. 7 — Sample of pressure data digitized with CAMAC system (1 PSI = 6.895 kPa)

was noticed that for these complex events the maximum pressures at stations 1, 2, and 8 were approximately equal, but at the same time they were substantially larger than those at the remaining stations (3 to 7). For single pressure rise events, the pressure rise at all stations was essentially equal. It is believed that the pressure readings were affected by thermal sensitivity of the gauges or by some other phenomena not understood by the author. For these reasons, the remainder of this report will concentrate on pressure measurements at side-on stations 3 through 7 (and at station 8 for single pressure rise events).

Ethylene-Air Tests

Twenty-one ethylene-air baseline tests were conducted. These tests are summarized in Table 3. In this table, the gas concentrations, the average side-on pressure at stations 3 to 7, and the standard deviation for the side-on pressure are given. Also listed is the reflected pressure at station 8 and the ratio of the side-on pressure to the reflected pressure (PR/PSO). The initial pressure rise, the impulse averaged over stations 3 to 7, and an indication of the type of reaction that developed complete the table. The variation in the average side-on pressure with the ethylene concentration is shown in Fig. 8. From this figure, the flammability limits for ethylene and air in the apparatus can be seen to be from 2% to about 16%. The lower explosive limit is in good agreement with the data in Table 1; however, the upper explosive limit from our experiments is lower than the literature value of 34%. Presumably the difference lies in the mode of ignition, the orientation of the vessel, and the vessel geometry.

No deflagrations were developed in the ethylene-air tests; however, for concentrations between 5.2% and 8.7% ethylene, very strong combustion reactions developed. Side-on pressures in these tests never exceeded 5.3 atm (537 kPa) and pressure rise rates were below 4200 psi/s (29 Mpa/s). It is interesting to note that the variation in the pressure rise rate with the ethylene concentration follows the same trend as the side-on pressure (see Fig. 9). The region of strong combustion reactions between 5.2% and 8.7% ethylene is also apparent in this figure.

As described in the introductory comments in this section, many different kinds of reactions, as exemplified by the shape of the pressure trace, were obtained. This is shown graphically in Fig. 10, where the pressure traces at station 5 for seven different ethylene-air tests are plotted. At ethylene concentrations between 10.4% and 15.6% and in the vicinity of 4.5% this kind of complicated pressure history was encountered. At all other concentrations, normal closed volume combustion events were observed. On Figs. 8 and 9 these multiple-rise events are indicated with symbols which are partially filled in, while open symbols are for normal combustion events. Apparently the multiple-rise events represent a transition concentration band between marginal and strong combustion events.

Ethylene-NF₃-Air Tests

Twenty-eight tests were conducted with NF₃-enriched ethylene-air mixtures. These tests are summarized in Table 4. The effect of adding NF₃ to ethylene-air mixtures is to increase substantially the severity of the reaction over what could be expected without NF₃ present in the mixture. This can be clearly seen in Figs. 11 and 12, where the side-on pressure and the pressure rise rate are plotted as a function of the ethylene concentration. In Fig. 11, five curves and in Fig. 12 four curves represent different levels of NF₃ present in the mixture before ignition. The 0% curves were obtained from the ethylene-air baseline tests and were transferred from Figs. 8 and 9. As the NF₃ concentration is increased, the side-on pressure increases uniformly, except at the very low ethylene concentrations, where only small differences in the side-on pressure were detected. Thus, the lower flammable limit appears to be relatively insensitive to the NF₃ concentration. This result is similar to that obtained with ethylene-oxygen and ethylene-nitrous oxide mixtures, as presented in Table 1. The lower flammable limit of ethylene in air, NF₃ and air, oxygen, or nitrous oxide is relatively constant, between 1.9% and 3.0%.

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Table 3 — Summary of Ethylene-Air Tests (1 PSI = 6.895 kPa; 1 ATM = 101.3 kPa)

TEST	C2H4 %	NF3 %	CF3BR %	SIDE-ON PRESSURE (PSI)	REFLECTED PRESSURE (PSI)	PRESSURE RATIO PR/PD (ATM)	PRESSURE RISE RATE (PSI/S)	IMPULSE (PSI-S)	REACTION
14	1.5	0.0	0.0	0.70 +/- 0.05	0.70	1.00	34.7	0.10	NO REACTION
15	2.6	0.0	0.0	0.70 +/- 0.22	7.06	1.33	105.5	1.10	COMBUSTION
13	3.1	0.0	0.0	5.30 +/- 0.00	29.27	1.74	372.5	8.20	COMBUSTION *
16	4.5	0.0	0.0	16.80 +/- 0.81	59.16	1.12	2034.2	6.20	COMBUSTION
11	5.2	0.0	0.0	52.60 +/- 3.07	72.31	1.01	4154.5	5.90	COMBUSTION
17	7.2	0.0	0.0	71.30 +/- 5.12	69.68	0.95	2755.3	6.60	COMBUSTION
18	7.8	0.0	0.0	66.60 +/- 2.41	71.23	1.09	3476.5	7.40	COMBUSTION
08	6.6	0.0	0.0	65.30 +/- 1.87	73.90	1.13	1989.0	12.10	COMBUSTION
03	8.7	0.0	0.0	65.30 +/- 3.09	78.09	1.19	718.1	15.90	COMBUSTION
02	9.1	0.0	0.0	65.70 +/- 3.20	69.00	1.28	460.2	16.20	COMBUSTION *
40	10.4	0.0	0.0	54.10 +/- 1.23	43.41	1.68	181.8	18.20	COMBUSTION *
19	11.2	0.0	0.0	25.80 +/- 1.04	49.20	1.39	331.8	15.70	COMBUSTION *
12	11.8	0.0	0.0	6.10 +/- 0.65	17.61	2.89	41.1	12.30	COMBUSTION *
05	14.1	0.0	0.0	32.80 +/- 0.76	49.02	1.49	326.8	15.30	COMBUSTION *
41	15.2	0.0	0.0	13.10 +/- 1.21	32.87	2.51	77.9	17.90	COMBUSTION
20	15.6	0.0	0.0	2.70 +/- 0.40	3.93	1.46	26.4	0.00	COMBUSTION
04	16.0	0.0	0.0	-----	-----	-----	-----	-----	NO REACTION
09	22.3	0.0	0.0	-----	-----	-----	-----	-----	NO REACTION
10	21.3	0.0	0.0	-----	-----	-----	-----	-----	NO REACTION
07	21.6	0.0	0.0	-----	-----	-----	-----	-----	NO REACTION
06	24.9	0.0	0.0	-----	-----	-----	-----	-----	NO REACTION

*—MULTIPLE PRESSURE RISE EVENT

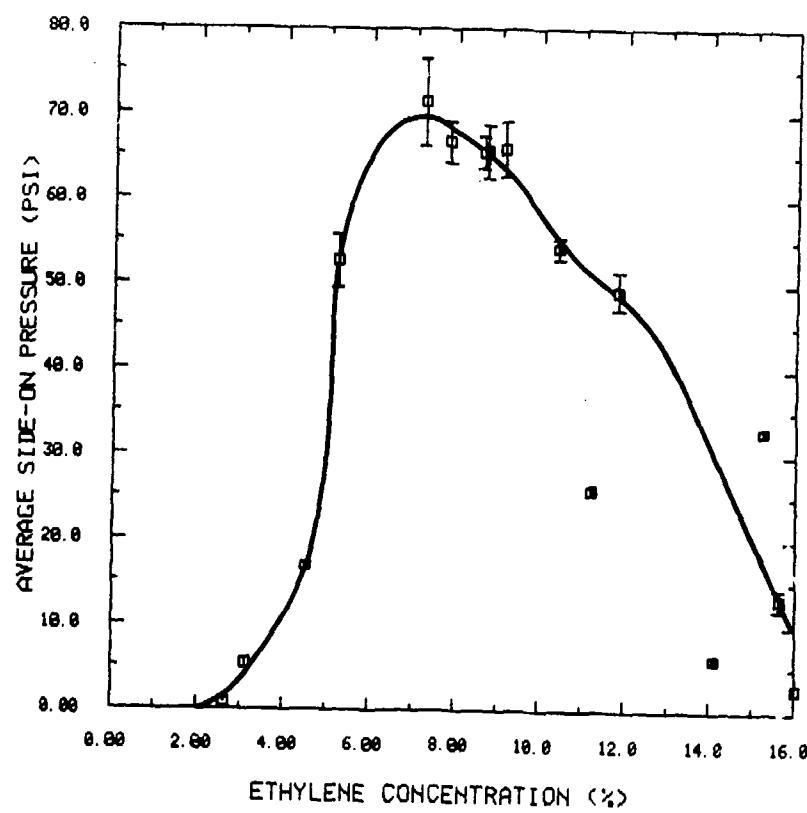


Fig. 8 — Side-on pressure as a function of the ethylene-air concentration

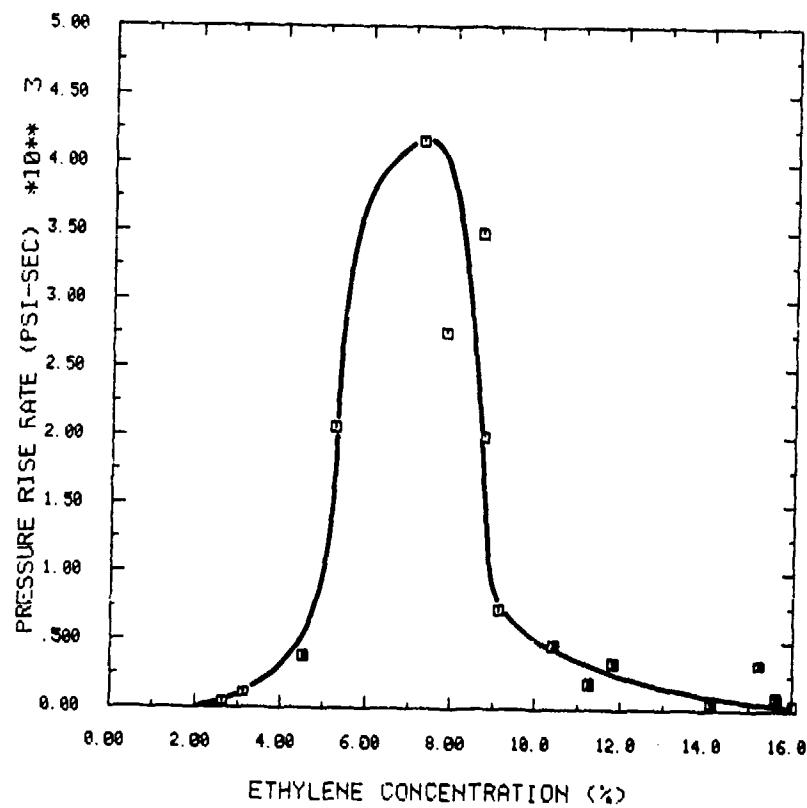


Fig. 9 — Initial pressure rise rate as a function of the ethylene-air concentration

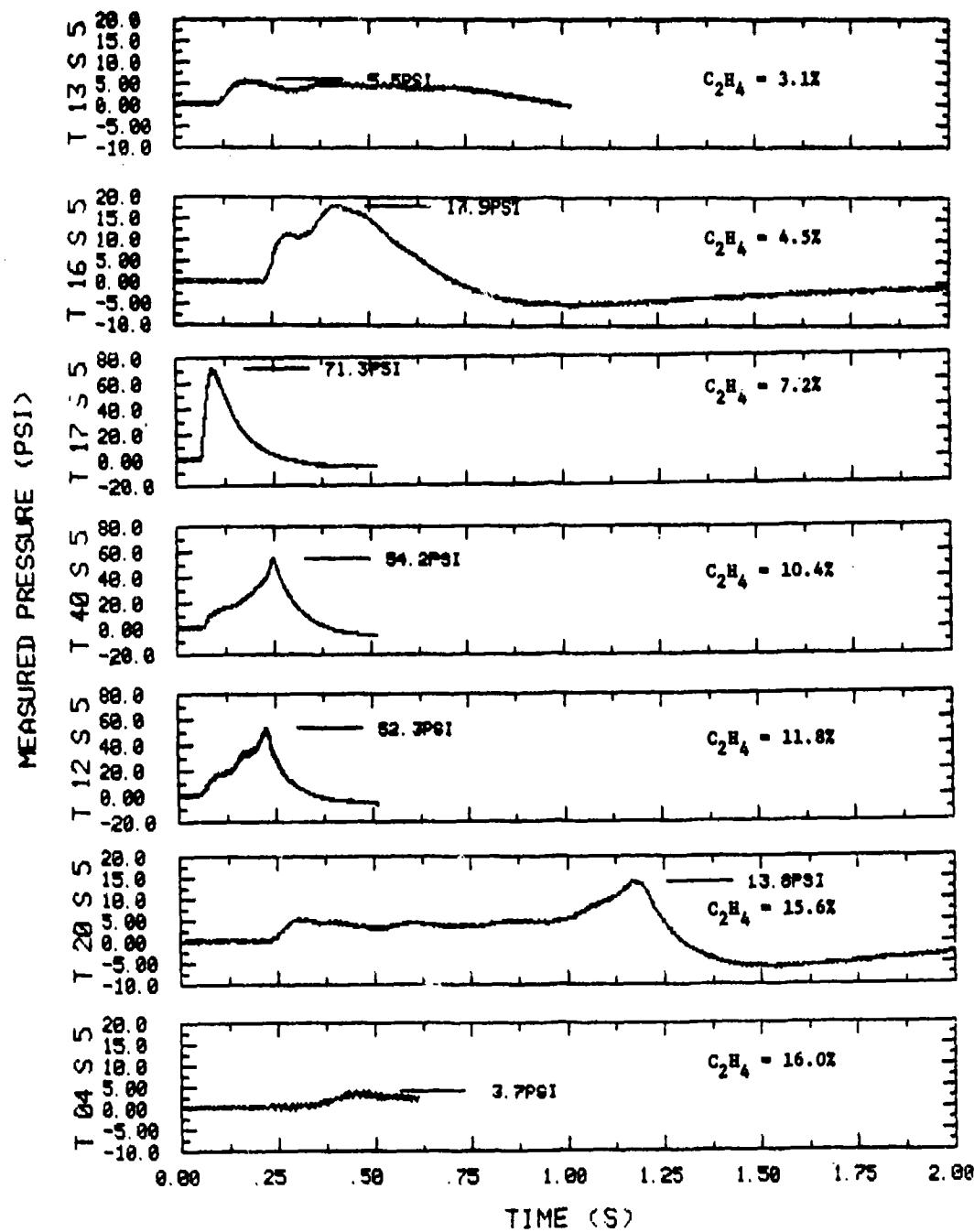


Fig. 10 — Comparison of pressure time histories obtained at different ethylene concentrations

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Table 4 — Summary of Ethylene- NF_3 -Air Tests (1 PSI = 6.895 kPa; 1 ATM = 101.3 kPa)

TEST	C ₂ H ₄ %	NF ₃ %	CF ₃ Br %	SIDE-ON PRESSURE (PSI)	REFLECTED PRESSURE (PSI)	PRESSURE RATIO PR/PSD (ATM)	PRESSURE RISE RATE (PSI/S)	IMPULSE (PSI- s)	REACTION
64	2.7	7.2	2.6	25.80 +/- 1.27	46.37	1.80	124.1	14.00	COMBUSTION
50	2.0	3.0	3.7	—	—	—	—	—	NO REACTION
65	5.6	5.4	2.5	83.10 +/- 3.81	88.00	1.06	1954.3	9.00	COMBUSTION
69	8.2	6.5	2.7	107.40 +/- 5.93	109.85	1.00	8014.8	4.50	DEFLAGRATION
66	10.0	8.9	2.7	131.40 +/- 5.14	137.57	1.05	17351.2	2.70	DEFLAGRATION
53	2.8	6.7	3.2	12.40 +/- 1.11	34.76	2.80	77.0	23.00	COMBUSTION
51	6.0	3.8	5.2	82.70 +/- 4.05	93.75	1.13	893.6	11.70	COMBUSTION
68	8.3	6.9	6.7	98.00 +/- 6.61	103.62	1.06	3209.6	6.90	DEFLAGRATION
52	10.0	5.5	5.2	79.80 +/- 3.70	89.97	1.13	673.7	11.60	COMBUSTION
55	10.3	8.7	5.3	120.80 +/- 2.36	125.40	1.04	4050.6	8.10	DEFLAGRATION
54	9.1	12.1	7.7	97.90 +/- 5.90	104.22	1.06	3314.8	6.50	DEFLAGRATION
56	2.8	4.2	9.2	—	—	—	—	—	NO REACTION
60	2.8	7.1	11.0	11.40 +/- 0.61	32.24	2.83	41.9	16.90	COMBUSTION
61	3.1	7.8	9.4	12.80 +/- 1.42	35.31	2.76	86.7	21.90	COMBUSTION
57	6.1	4.7	9.9	—	—	—	—	—	NO REACTION
59	6.1	4.7	9.9	17.10 +/- 1.18	46.74	2.73	75.6	26.20	COMBUSTION
72	8.4	6.7	10.6	67.10 +/- 1.98	82.93	1.24	236.0	19.00	COMBUSTION
73	8.4	6.7	10.6	72.20 +/- 2.05	92.88	1.27	339.4	16.90	COMBUSTION
58	9.9	5.4	9.9	3.30 +/- 0.23	4.50	1.29	46.3	0.60	COMBUSTION
62	10.6	7.4	10.2	42.30 +/- 4.07	53.40	1.26	318.4	13.70	COMBUSTION
67	14.4	13.2	11.5	107.60 +/- 5.12	115.00	1.07	3943.1	6.70	DEFLAGRATION
70	4.3	12.2	15.7	9.80 +/- 2.27	29.03	2.96	20.4	17.00	COMBUSTION
71	4.3	12.2	15.7	12.70 +/- 2.82	35.85	2.82	91.4	21.40	COMBUSTION

*=MULTIPLE PRESSURE RISE EVENT

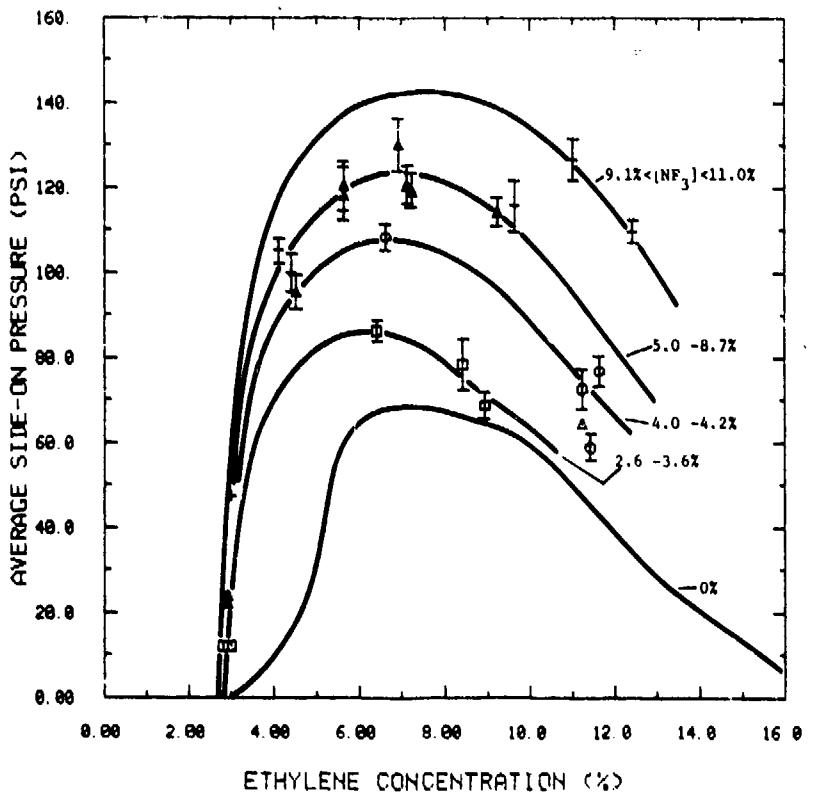


Fig. 11 - Side-on pressure curves for the ethylene-NF₃-air tests

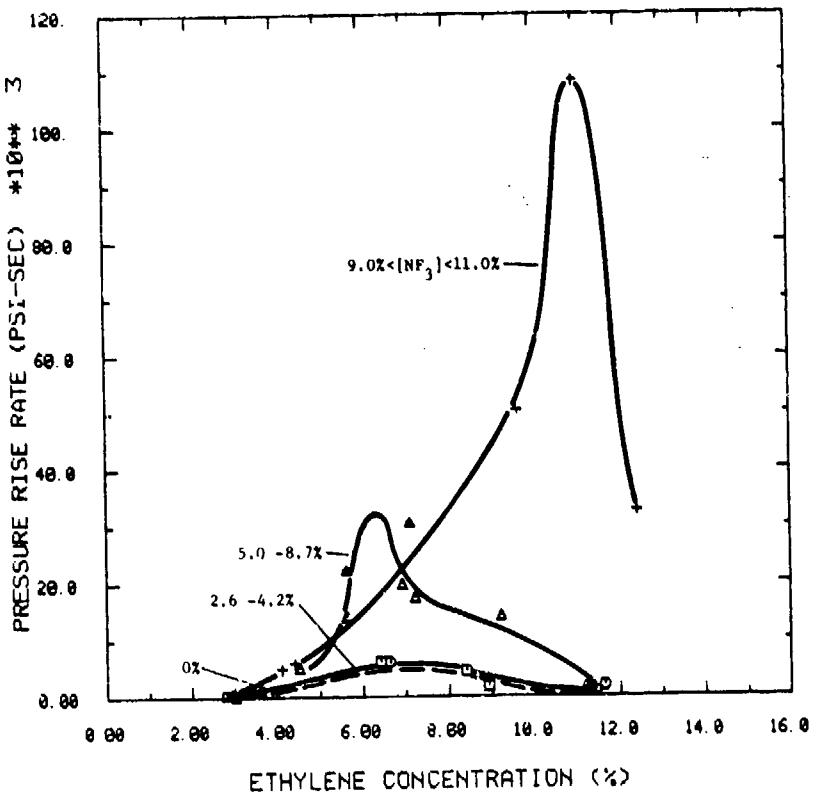


Fig. 12 — Pressure rise rate curves for the ethylene NF_3 -air tests

The side-on pressure curves exhibit similar trends regardless of the NF_3 concentration. The curves all show a maximum pressure between 6.0% and 8.0% ethylene. No substantial disparity between combustion and deflagration reactions is apparent from Fig. 11. The pressure rise rate curves, however, exhibit different trends: for high NF_3 concentrations above 5.0%, they have both deflagration and combustion reaction points. The deflagration reaction points fall on a narrow peak of the pressure rise rate curve. Unlike the side-on pressure data, the peak pressure rise rate occurs at different ethylene concentrations for different NF_3 concentrations. The peak pressure rise rate occurs between 5.5% and 7.0% ethylene for NF_3 in the range of 5.0% to 8.4% and between 5.6% and 12.5% for NF_3 in the range of 9.1% to 11.0%.

The effect of adding NF_3 to ethylene-air mixtures can be seen if we plot the maximum pressure developed at any ethylene concentration as a function of the amount of NF_3 enrichment. This plot is shown in Fig. 13. From this curve the maximum pressure appears to be a nearly linear function of the NF_3 concentration, with about 7.5 psi (51.7 kPa) increase in pressure for each additional percent of NF_3 enrichment.

The pressure data collected during this series of tests can be used to establish contours of equal pressure on a plot of NF_3 concentration vs ethylene concentration. Such a plot is given in Fig. 14. The contours were established by plotting the NF_3 and ethylene concentrations for each test conducted, sketching in lines that separated different pressure levels, e.g., 50 and 75 psi (345 and 517 kPa), and then drawing smooth contours following the trend of the sketched lines. In this curve the types of reactions that develop are indicated by different symbols. Deflagrations are represented by closed symbols, combustion events by half-closed symbols, multiple-rise events by one-quarter-closed symbols, and no reactions by open symbols. From these curves the regions where the different reaction types occur can be clearly seen.

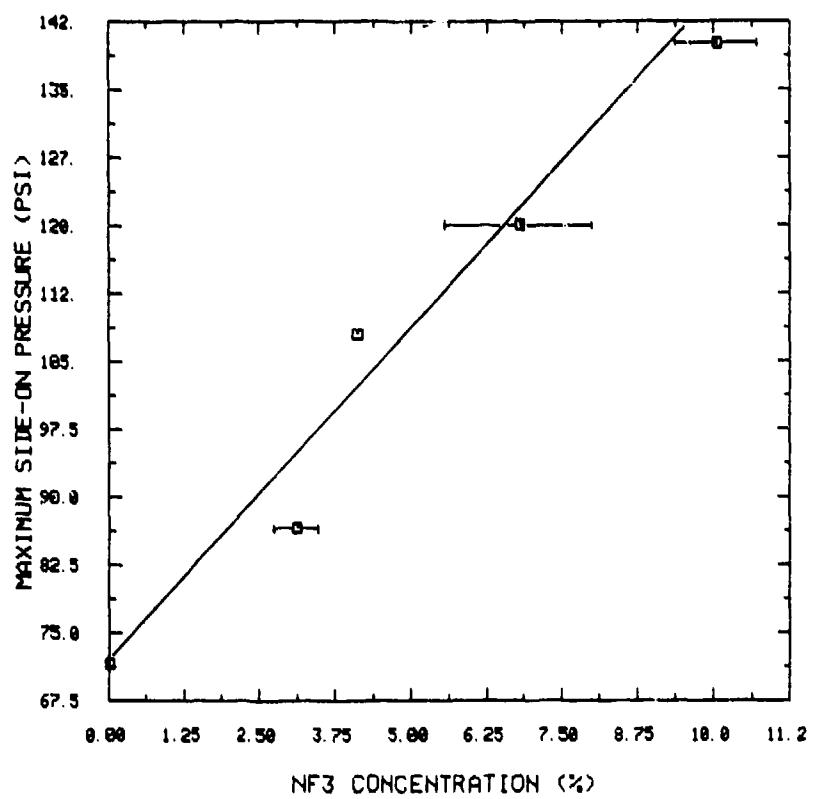


Fig. 13 — Effect of NF_3 concentration on maximum side-on pressure

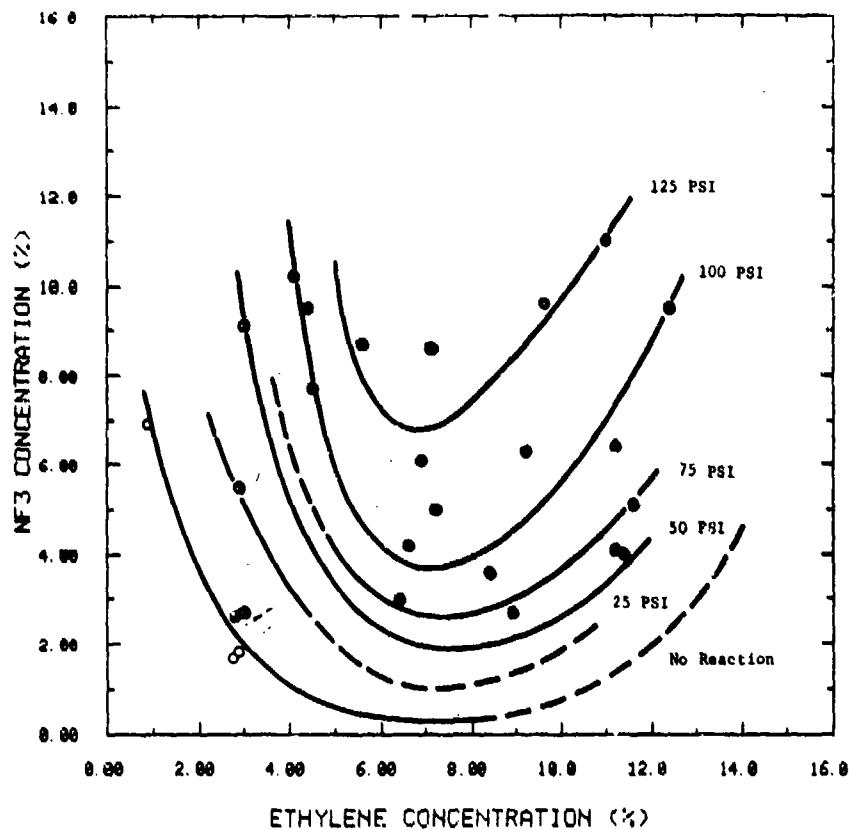


Fig. 14 — Constant-pressure contours derived from the ethylene-NF₃-air tests

As was the case for the ethylene-air baseline tests, many different kinds of reactions were encountered. The reactions ranged from weak combustion to multiple-rise combustion to strong combustion and deflagration events. This is shown in Fig. 15, where the pressure traces at station 5 for six tests with NF₃ concentrations in the range of 5.5% to 8.6% are given. At the low ethylene concentration present in Test 22, a multiple-rise event was encountered. As the ethylene concentration was increased to 4.5%, a conventional closed volume combustion pressure event was recorded. For ethylene concentrations in the range of 5.6% to 7.1%, deflagration reactions were encountered. Note the substantially higher pressure rise rate and the shorter overall duration of these events. At ethylene concentrations near 11%, normal combustion events were detected. Similar trends were encountered with the other NF₃ concentration ranges.

Ethylene-NF₃-Halon 1301-Air Tests

Twenty-three tests were conducted in which the effect of adding halon 1301 to the ethylene-NF₃-air mixtures was studied. These tests are summarized in Table 5. *The effect of adding halon to the gas mixture is to reduce the severity of the reaction below what would have been expected with no halon present.* This effect can be seen clearly in Figs. 16 and 17, where the side-on pressure and the pressure rise rate for tests with NF₃ in the range of 5.4% to 8.7% are plotted. In each figure, four curves are presented for different levels of halon 1301 present in the mixture prior to ignition. The 0% curve on each figure represents the trend obtained with no halon present and was transferred from the appropriate curve on Fig. 11 or Fig. 12. As the halon 1301 concentration is increased,

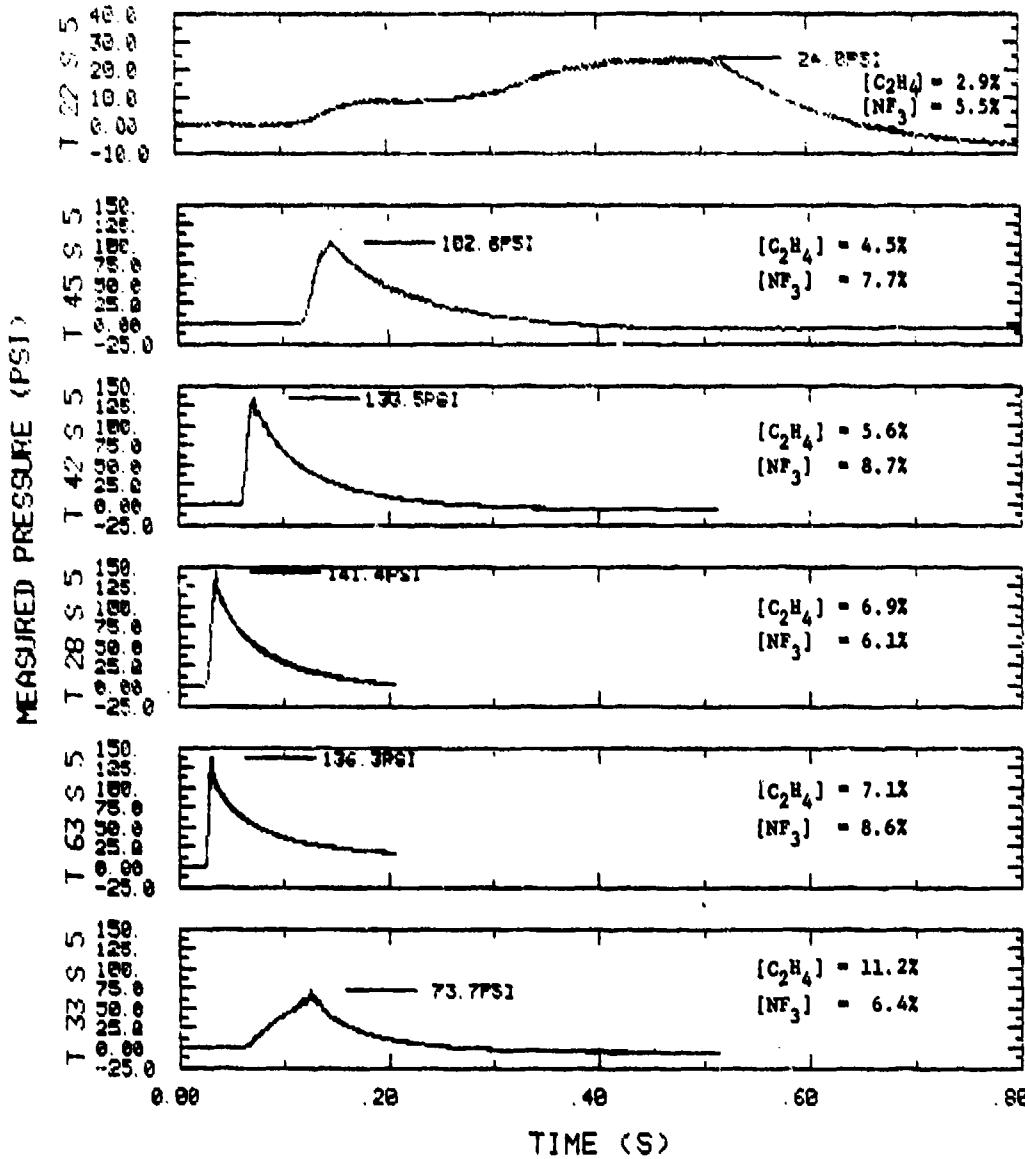
Fig. 15 — Comparison of pressure time histories obtained in the ethylene-NF₃-air test

Table 5—Summary of Ethylene-NF₃ Halon 1301-Air Tests (1 PSI = 6.895 kPa; 1 ATM = 101.3 kPa)

TEST	C ₂ H ₄ %	NF ₃ %	CF ₃ Br %	SIDE-ON PRESSURE (PSI)	REFLECTED PRESSURE (PSI)	PRESSURE RATIO PR/PRI (ATM)	PRESSURE RISE RATE (PSI/S)	IMPULSE (PSI-S)	REACTION
27	2.7	2.4	0.0	—	—	—	—	—	NO REACTION
25	3.0	2.7	0.0	—	—	—	—	—	NO REACTION
30	2.8	2.4	0.0	11.90 +/-	0.35	31.91	2.68	150.4	11.40 COMBUSTION
21	3.0	2.7	0.0	12.10 +/-	0.50	31.49	2.60	26.9	11.00 COMBUSTION
37	6.4	3.0	0.0	86.20 +/-	2.47	90.40	1.05	6073.6	6.00 COMBUSTION
46	9.4	3.6	0.0	78.10 +/-	4.06	88.60	1.13	4275.2	6.50 COMBUSTION
48	0.9	2.7	0.0	68.70 +/-	3.10	77.00	1.12	1713.2	0.90 COMBUSTION
26	6.6	4.2	0.0	108.20 +/-	3.30	0.00	0.00	2916.0	6.60 COMBUSTION
32	11.2	4.1	0.0	72.40 +/-	4.59	80.00	1.10	1364.2	7.50 COMBUSTION
38	11.4	4.0	0.0	58.80 +/-	3.00	70.00	1.19	803.6	7.60 COMBUSTION
36	1.0	6.8	0.0	—	—	—	—	—	NO REACTION
22	2.7	5.5	0.0	24.00 +/-	0.89	46.32	1.92	180.2	20.10 COMBUSTION
24	2.7	5.5	0.0	22.20 +/-	0.72	44.09	1.49	211.1	18.50 COMBUSTION
45	4.5	7.7	0.0	95.30 +/-	4.11	104.70	1.10	5016.0	6.20 COMBUSTION
42	5.6	8.7	0.0	120.30 +/-	3.89	125.00	1.04	22194.9	3.30 DEFLAGRATION
43	5.6	8.7	0.0	118.40 +/-	6.37	125.20	1.06	14002.0	3.70 DEFLAGRATION
23	6.9	6.1	0.0	130.00 +/-	6.27	0.00	0.00	19753.4	2.00 DEFLAGRATION
63	7.1	8.6	0.0	120.60 +/-	4.48	122.00	1.01	30996.9	1.60 DEFLAGRATION
27	7.2	5.0	0.0	119.20 +/-	4.00	115.10	0.97	17410.6	2.50 DEFLAGRATION
34	9.2	6.3	0.0	114.30 +/-	3.38	112.00	0.93	16020.8	2.70 DEFLAGRATION
33	11.2	6.4	0.0	64.30 +/-	1.59	71.33	1.41	1164.0	8.30 COMBUSTION
47	11.6	5.1	0.0	76.70 +/-	3.74	82.00	1.07	1724.1	7.70 COMBUSTION
23	3.0	9.1	0.0	47.50 +/-	1.95	60.00	1.26	633.1	11.50 COMBUSTION
37	4.1	10.2	0.0	104.90 +/-	2.73	108.00	1.03	4765.5	5.30 COMBUSTION
31	4.4	9.5	0.0	99.70 +/-	4.53	107.40	1.08	5762.4	5.97 COMBUSTION
44	9.6	9.6	0.0	115.70 +/-	6.00	112.50	0.97	50199.4	1.42 DEFLAGRATION
35	11.0	11.0	0.0	126.50 +/-	4.90	145.00	1.15	108055.7	0.90 DEFLAGRATION
49	12.4	9.5	0.0	107.40 +/-	2.63	112.04	1.02	32488.4	1.00 DEFLAGRATION

*—MULTIPLE PRESSURE RISE EVENT

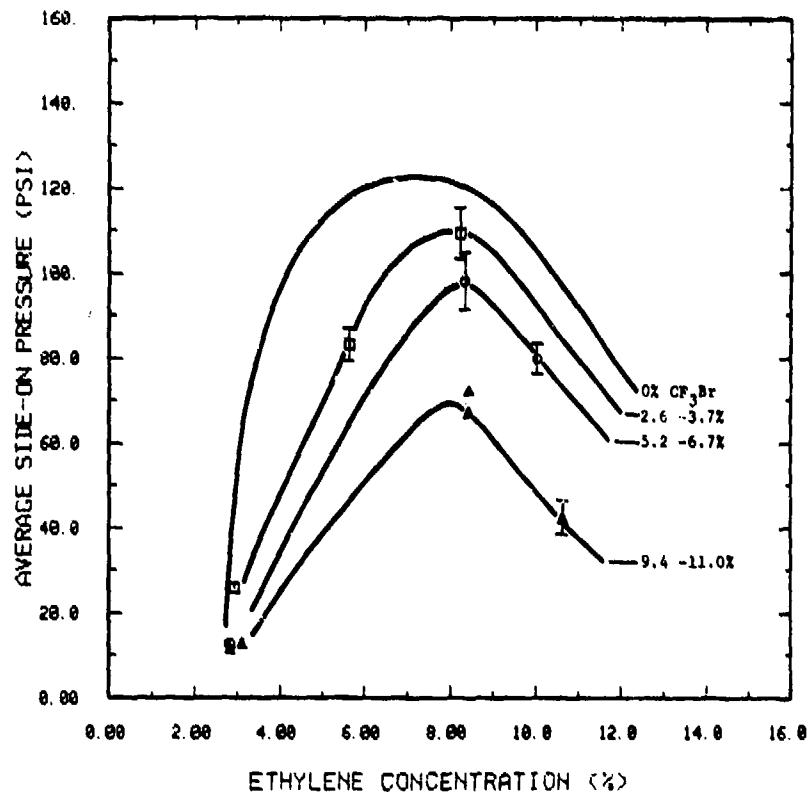


Fig. 16 — Side-on pressure curves for the ethylene- NF_3 -halon 1301-air tests

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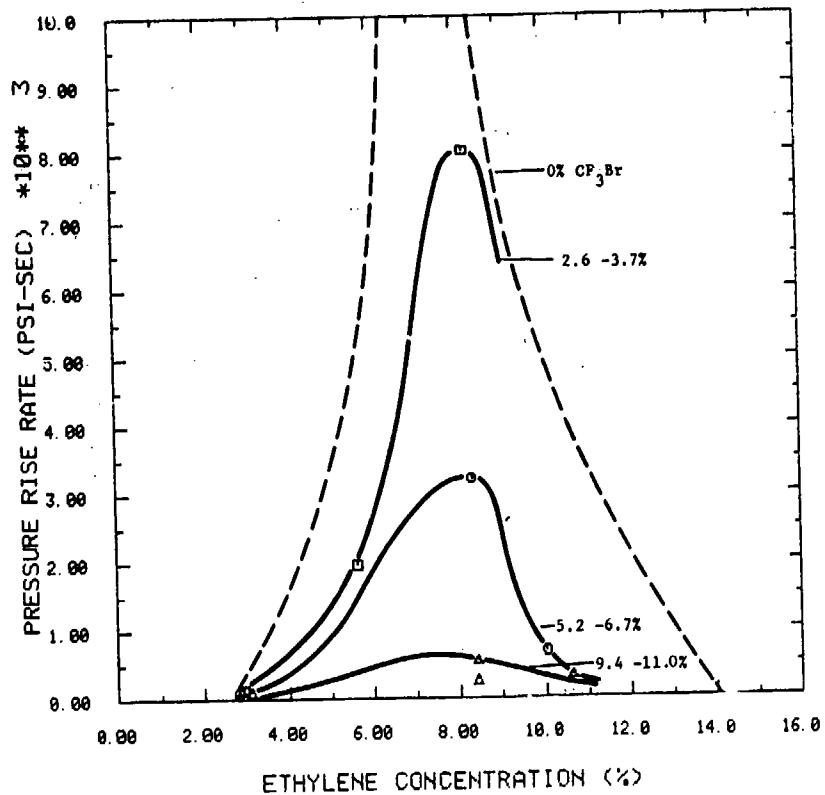


Fig. 17 — Pressure rise rate curves for the ethylene-NF₃-halon 1301-air tests

the side-on pressure is uniformly reduced over what would be expected without the presence of halon. The curves roughly parallel each other, with the peak pressure occurring in the range of 6.0% to 8.0% ethylene in each case. The presence of the fire retardant significantly reduces the pressure rise rate, as is seen in Fig. 18. Again, as long as no deflagration events occur, the trend of the pressure rise rate curve is similar to that of the side-on pressure curves. Deflagration reactions occur in a narrow peak, in this case between 8.0% and 9.0%, whereas with no halon present the peak occurred near 6.0%. Pressure rise rates for the halon experiments with NF_3 concentrations in the range of 5.4% to 8.7% were always below 8000 psi/s (55 MPa/s) as compared with rise rates exceeding 20,000 psi/s (138 MPa/s) with no halon present.

The effect of adding halon 1301 to the ethylene- NF_3 -air mixtures can also be seen if we plot the maximum pressure developed at any ethylene concentration as a function of the halon concentration as in Fig. 18. For halon addition in the range of 0% to 6%, the pressure is reduced linearly with increasing halon concentration. For concentrations of halon above 6% the pressure appears to decay at a higher rate. Halon concentrations near 10% show a reduction of nearly half the reaction intensity as represented by the side-on pressure.

The pressure data collected during the halon test series were used to develop a series of contours of equal pressure similar to the one developed for NF_3 tests. These contours are presented in Figs. 19, 20, and 21 for low halon concentrations (2.5% to 3.7%), medium concentrations (5.2% to 6.7%), and high concentrations (9.2% to 11.5%). On these plots, combustion, multiple-rise, deflagration, and no-reaction events are indicated by the symbol convention introduced

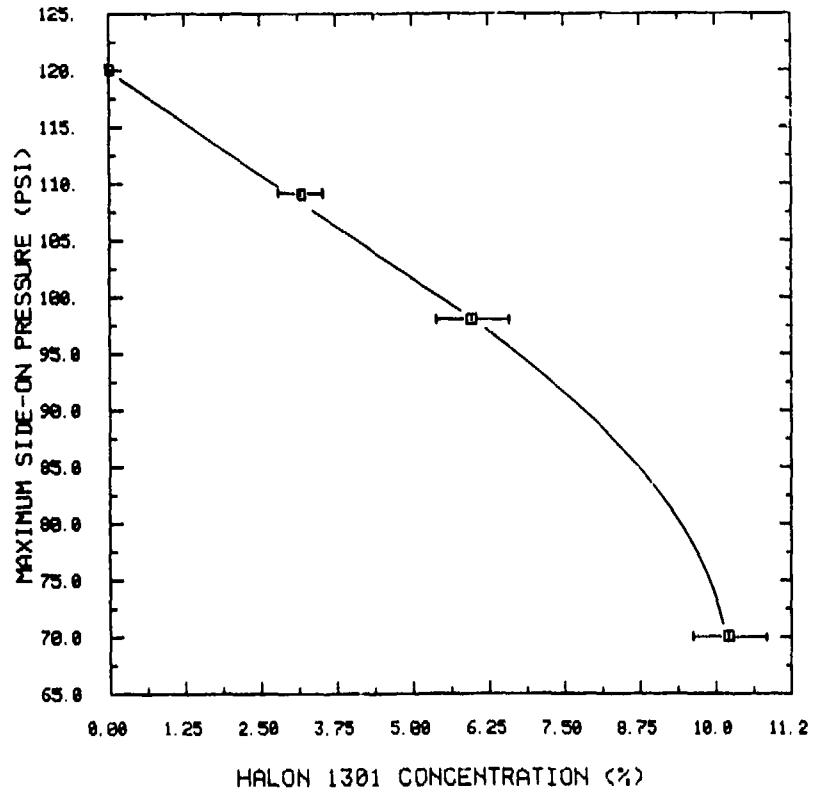


Fig. 18 — Effect of halon 1301 concentration on maximum side-on pressure

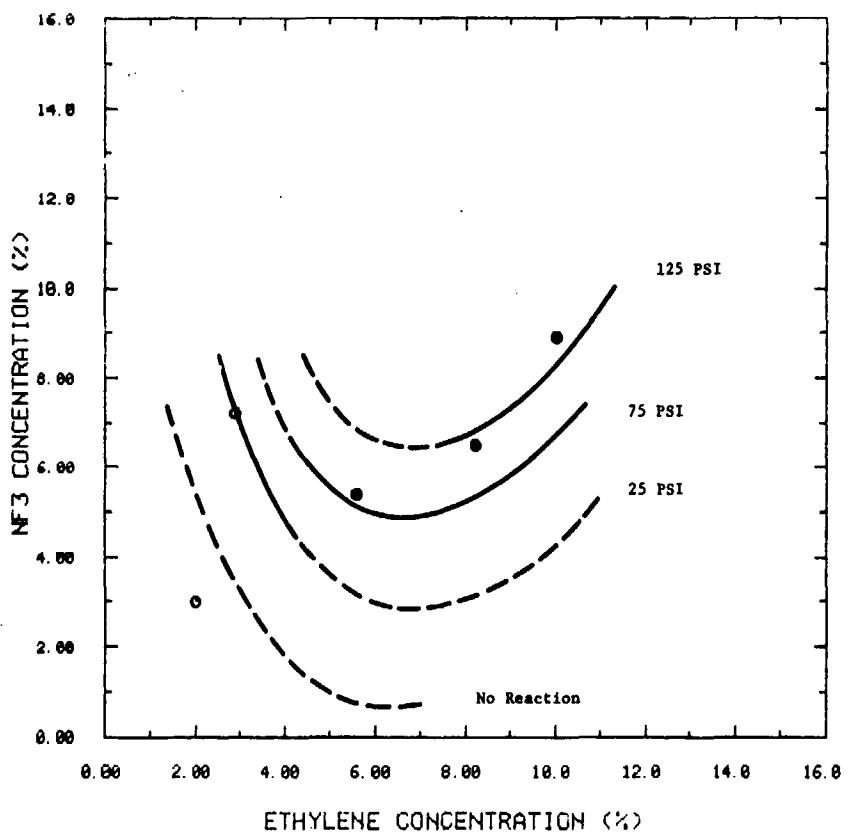


Fig. 19 — Constant-pressure contours derived from the low-halon-concentration tests

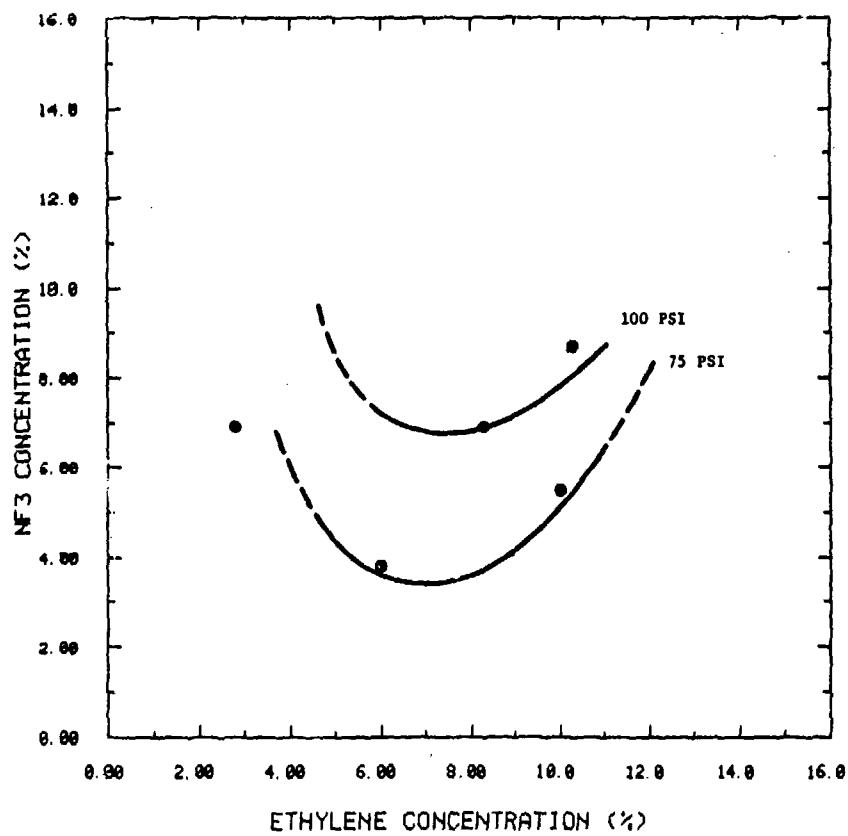


Fig. 20 — Constant-pressure contours derived from the medium-halon-concentration tests

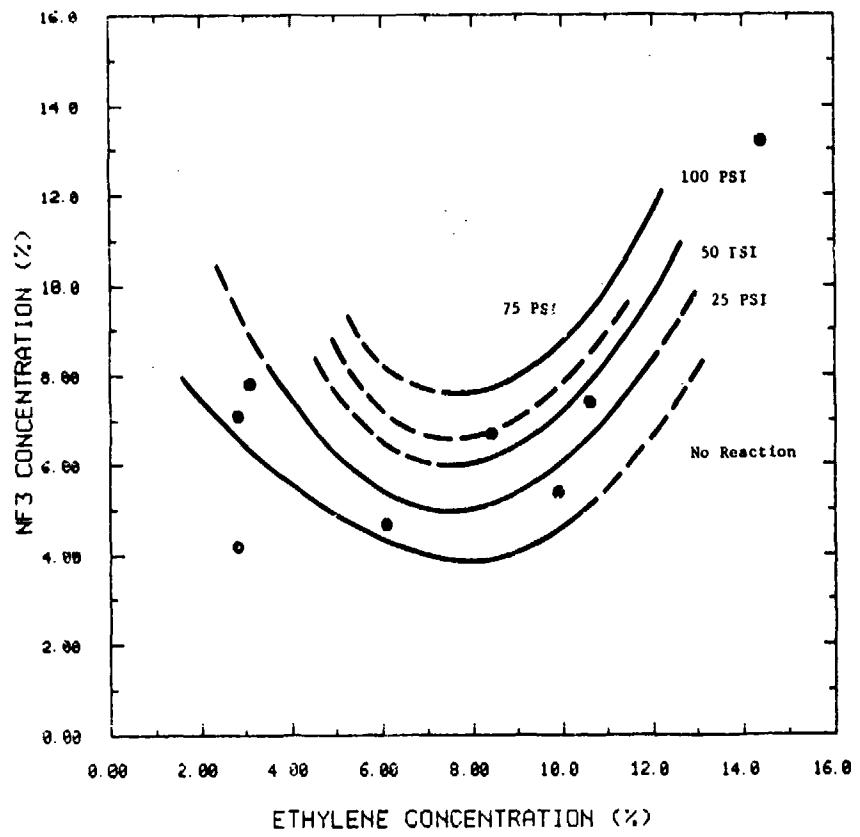


Fig. 21 — Constant-pressure contours derived from the high-halon-concentration tests

on page 19 (and Fig. 14) for the NF_3 test series. These plots again demonstrate that the reaction intensity is reduced by the use of the halon 1301 fire retardant.

Analysis of Reaction Products

The reaction products for tests 70 through 73 were analyzed using a mass spectrometer. The gas analysis report is given in Table 6. The main conclusion from these analyses was that the bromine from the CF_3Br reacts with ethylene to produce dibromomethane,



Since we did only a product analysis, the pathway of this reaction is unknown. Additionally, HBr could be found, but it was not detectable with the analysis scheme used. The fluorine from the CF_3Br reaction is assumed to react to form HF and CF_4 , which have been found as the major fluorinated species in other NF_3 ethylene combustion tests we have studied [1]. No toxicity data for CH_2Br_2 were found; however, toxicity is expected to be similar to that of CH_3Br . The compound CH_3Br is toxic to rats at a concentration of 500 ppm for 3 h [12]. Therefore, HF is the principal toxic hazard in an NF_3 fire, even when it is inerted with CF_3Br .

Table 6 — Relative Abundance of Major Constituents

Compound	Relative Abundance			
	Sample 1	Sample 2	Sample 3	Sample 4
NF_3	0.05	0.04	0.45	0.31
CF_3Br	1	1	1	1
Cyanogen bromide	ND*	ND*	ND*	0.02
Unidentified				
C_6 hydrocarbon	1.5	0.3	0.94	0.71
Dibromomethane	0.34	0.27	0.06	0.69

*ND — not detected

SUMMARY AND CONCLUSIONS

The principal conclusions drawn from the test results obtained on this program are:

- Addition of NF_3 to ethylene-air mixtures increases the upper flammable limits and increases the severity of the reaction. The lower flammable limit is essentially unchanged. The maximum pressure developed in ethylene- NF_3 -air mixtures increases linearly with increased NF_3 enrichment.
- Halon 1301 was found to be an effective fire extinguishing agent for ethylene- NF_3 -air reactions. By use of the ICI cup burner, it was found that about 6.5% of halon 1301 is required to extinguish ethylene-air flames with no NF_3 enrichment. For each addition of 1% of NF_3 , an average of 0.88% additional halon is required for extinguishment. In the explosion tests, it was found that halon 1301 is capable of significantly reducing the severity of the reaction. For moderate

NF_3 concentrations, a reduction in reaction intensity of 50% can be achieved with the addition of 10% halon 1301.

- Two kinds of combustion reactions were observed during these tests. One type exhibited the normal closed volume combustion pressure trace. The other type was characterized by a normal pressure rise and decay followed by a subsequent unusually high amplitude pressure rise. These multiple-rise events were observed only near the rich and lean flammability limits.
- Mass spectrographic analysis of the reaction products revealed dibromomethane and an unidentified C_6 hydrocarbon in the reaction products.

RECOMMENDATIONS FOR FUTURE STUDIES

The results of this work clearly show that halon 1301 is effective in reducing the danger of combustion and explosion in an NF_3 -enriched environment. To determine the amount of halon 1301 required for an actual installation, a failure-mode-effects analysis must be carried out for the design. In particular, the analysis should identify the volume of the compartment and quantity of NF_3 likely to be released. Using these data, a full-size model could be constructed and validation of the inerting by halon 1301 verified. The model would be constructed of a frame having polyethylene sheeting for walls. This would be inexpensive and would allow good visual observation of the experiments.

Additionally, experiments should be designed to verify that a suitable leak detector for NF_3 is available. If it is not, then various analytical methods should be studied to determine what approach is best for this task. Without a leak detector, release of halon 1301 would be under manual control, which would increase the risk due to delayed activation of the inerting system.

ACKNOWLEDGMENT

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Appendix

ABBREVIATIONS

CAMAC	Computer automated measurement and control
CH_2Br_2	Dibromomethane
CH_3Br	Bromomethane
C_2H_4	Ethylene
EBW	Exploding bridgewire
Halon 1301	CF_3Br (Bromo trifluoromethane)
HBr	Hydrogen bromide
HF	Hydrogen fluoride
ICI	Imperial Chemical Industries
NACL	Navy ARPA Chemical Laser
NAVSEA	Naval Sea Systems Command
NF_3	Nitrogen trifluoride
NRL	Naval Research Laboratory
PCB	Piezoelectronics Inc. symbol
SWRI	Southwest Research Institute